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Technical Report No. 2

METHACRYLAMIDE COPOLYMER RESISTS
FOR ELECTRON BEAM LITHOGRAPHY

by

Y. M. N. Namaste, S. K. Obendorf, and F. Rodriguez

Prepared for presentation at the
Santa Clara Symposium on Microlithography
Conference 631: Advances in Resist Technology and Processing III
Society of Photo-Optical Instrumentation Engineers
Santa Clara, CA, March 9-14, 1986

Olin Hall, Cornell University
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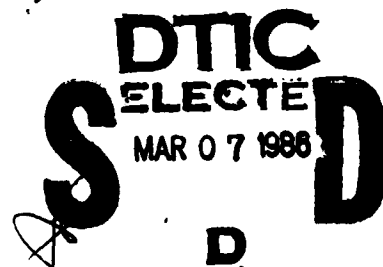
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Methacrylamide copolymer resists for electron beam lithography

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Abstract

Polymethacrylamide (PMAAm) and copolymers of MAAm with methyl methacrylate were
synthesized and evaluated for their applicability to electron beam lithography. The
sensitivity of PMAAm has previously been reported as less than 1 $\mu C/cm^2$, with thermal
stability at temperatures up to 330°C.[1] Despite these claims, further lithographic
evaluation of this resist system is apparently absent from the literature. This research
was conducted to further investigate the lithographic performance of these resists and to
determine their sensitivity using current definitions.

Using PMAAm homopolymer (Mw = 8.1 x 10⁴), with a 15 minute prebake at 200°C, the
lithographic results were much poorer than expected. Patterns exposed to doses of
10 $\mu C/cm^2$ or lower could not be developed using water as the developing solvent. Forced
developing with Na_2SiO_3 solution (pH-10) developed lower doses than water, but much
greater thinning was observed. An unexposed thinning of 10% occurred when developing
exposures of 15 $\mu C/cm^2$ with water, and 40 $\mu C/cm^2$ with Na_2SiO_3 solution (20 KV). Swelling
of the unexposed polymer and some adhesion problems were observed.

The high sensitivity previously reported for PMAAm[1] can not be attributed solely to
chain scission efficiency (Gs), which has been reported to be only 1.5 times that of PMMA[2]
(Gs determined by Y-irradiation). An induction period in the dissolution of unexposed
polymer has also been sugggested as contributing to the sensitivity of this resist. In
the present work, dissolution induction periods were observed with laser interferometry
for the unexposed films, but the magnitude of these induction periods could not account
for a large enhancement of sensitivity. Imide crosslink formation may have been
responsible for the previously reported sensitivity of PMAAm.[1] In the present work, imide
formation was not observed, either after prebaking coated wafers at 180 to 240°C or
heating of polymer solutions for 7 hours at 80°C. Apparently, the conditions for useful
formation of imide are difficult to reproduce.

Because of the problems encountered with the PMAAm homopolymer during lithography, a
series of MMA - MAAm copolymers were synthesized and evaluated as potential resists.
These copolymers eliminated the problems of swelling and poor solubility of the
homopolymer, but sensitivity enhancement over that of PMMA was minimal.

Introduction

The search continues for a highly sensitive positive electron beam resist. Many
researchers are presently working toward the goal of producing a resist with sensitivity
in the 1 $\mu C/cm^2$ range, which concurrently exhibits good thermal stability and reactive ion
etch resistance.

Several years ago, workers in Japan reported on polymethacrylamide (PMAAm) as a
positive electron resist. They reported a sensitivity of about 0.6 $\mu C/cm^2$ at an
accelerating voltage of 20 KV and thermal stability at temperatures up to 330°C.[1]
Although this work is referenced in numerous review articles, there is apparently no
further work reported on this resist since the original work was completed. This is
surprising, given the excellent sensitivity originally reported.

In the present work, methacrylamide polymers are synthesized and evaluated with the
intention of reproducing previous sensitometry and then improving on the resist system by
evaluating similar polymers and copolymers of methacrylamide. The factors influencing the
sensitivity are investigated, including chain-scission efficiency, prebake conditions,
developing systems, and dissolution induction periods.

Experimental

Polymethacrylamide (PMAAm) homopolymers and methacrylamide - methyl methacrylate
copolymers (MAAm-MMA) were synthesized by free-radical polymerization, initiated with
potassium persulfate. The polymerizations were carried out in mixtures of water and

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1. REPORT NUMBER 2	2. GOVT ACCESSION NO. AD-A1... 0-3	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) METHACRYLAMIDE COPOLYMER RESISTS FOR ELECTRON BEAM LITHOGRAPHY		5. TYPE OF REPORT & PERIOD COVERED Technical Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Y.M.N. Namaste, S.K. Obendorf, and F. Rodriguez		8. CONTRACT OR GRANT NUMBER(s) N00014-85-K-0474
9. PERFORMING ORGANIZATION NAME AND ADDRESS Cornell University Ithaca, NY 14853		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Chemistry Office of Naval Research Arlington, VA 22217		12. REPORT DATE February 28, 1986
		13. NUMBER OF PAGES 7
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/ DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

Abstracts Distribution List, 356A/413-2.
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18. SUPPLEMENTARY NOTES

To be presented at "Advances in Resist Technology and Processing III," part of the Santa Clara Symposium on Microlithography sponsored by the Soc. of Photo-Optical Instrumentation Engineers, March 9-14, 1986, Santa Clara, CA.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Poly(methacrylamide)
Microlithography
Electron beam resist
Methacrylamide copolymer

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Polymethacrylamide (PMAAm) and copolymers of MAAm with methyl methacrylate were synthesized and evaluated for their applicability to electron beam lithography. The sensitivity of PMAAm has previously been reported as less than 1 $\mu C/cm^2$, with thermal stability at temperatures up to 330°C.[1] Despite these claims, further lithographic evaluation of this resist system is apparently absent from the literature. This research was conducted to further investigate the lithographic performance of these resists and to determine their sensitivity using current definitions.

Using PMAAm homopolymer (Mw = 8.1×10^6), with a 15 minute prebake at 200°C, the lithographic results were much poorer than expected. Patterns exposed to doses of 10 $\mu C/cm^2$ or lower could not be developed using water as the developing solvent. Forced developing with Na_2SiO_3 solution (pH = 10) developed lower doses than water, but much greater thinning was observed. An unexposed thinning of 10% occurred when developing exposures of 15 $\mu C/cm^2$ with water, and 40 $\mu C/cm^2$ with Na_2SiO_3 solution (20 KV). Swelling of the unexposed polymer and some adhesion problems were observed.

The high sensitivity previously reported for PMAAm[1] cannot be attributed solely to chain scission efficiency (Gs), which has been reported to be only 1.5 times that of PMMA[2] (Gs determined by γ-irradiation). An induction period in the dissolution of unexposed polymer has also been suggested as contributing to the sensitivity of this result. In the present work, dissolution induction periods were observed with laser interferometry for the unexposed films, but the magnitude of these induction periods could not account for a large enhancement of sensitivity. Imide crosslink formation may have been responsible for the previously reported sensitivity of PMAAm.[1] In the present work, dissolution induction periods were observed with laser interferometry for the unexposed films, but the magnitude of these induction periods could not account for a large enhancement of sensitivity. Imide crosslink formation may have been responsible for the previously reported sensitivity of PMAAm.[1] In the present work, imide formation was not observed, either after prebaking coated wafers at 180 to 240°C or heating of polymer solutions for 7 hours at 80°C. Apparently, the conditions for useful formation of imide are difficult to reproduce.

Because of the problems encountered with the PMAAm homopolymer during lithography, a series of MMA-MAAm copolymers were synthesized and evaluated as potential resists. These copolymers eliminated the problems of swelling and poor solubility of the hompolymer, but sensitivity enhancement over that of PMMA was minimal.

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Table I - Polymer synthesis

Polymer	MAAm (g)	MMA (g)	Water (ml)	Acetone (ml)	Initiator (g)	Temperature (°C)	Time (hrs.)	Recovery (g)
PMAAm (low MW)	25	--	45	130	0.05	60	4	17
PMAAm (high MW)	25	--	22.5	65	0.018	55	7	17
P(MMA-co-21% MAAm)	7	18	22.5	65	0.018	55	2	2.8
P(MMA-co-49% MAAm)	15	10	22.5	65	0.018	55	2.25	4.1
P(MMA-co-71% MAAm)	20	5	22.5	65	0.018	55	1	1.6

acetone. Details of the polymerizations are listed in Table I. Two molecular weights of the PMAAm homopolymer were synthesized, and three compositions of the MAAm-MMA copolymers were synthesized. The homopolymers synthesized are soluble in formamide, and in aqueous solutions of acetic acid and urea. The calculated compositions and measured intrinsic viscosities of the MAAm-MMA copolymers are presented in Table II. The copolymer compositions have been confirmed using IR spectroscopy.

Table II - Copolymer compositions and viscosities

Mol. Fraction MAAm (calculated)	Intrinsic Viscosity [n], dl/g 30°C	Solvent
1.00	2.5	Acetic acid (glacial)
0.71	2.8	Acetic acid (0.707 wt. fraction)
0.49	3.1	Acetic acid (glacial)
0.21	0.7	Chloroform

Intrinsic viscosities of the PMAAm homopolymers, determined in 8 M urea at 30°C, are 0.405 and 0.90 dl/g. Using a relationship reported by Titkova[*] for 8 M urea, these viscosities correspond to weight-average molecular weights (Mw) of 2.3×10^5 and 8.1×10^5. The higher molecular weight PMAAm is not soluble in water at any temperature. The lower molecular weight PMAAm is soluble in water only when heated to 90°C, but this polymer reprecipitates upon cooling. In the present work, the best solvent identified for the homopolymer is a 40% aqueous solution of acetic acid, which is used for casting the homopolymer films.

Matsuda and coworkers[*] reported on a PMAAm polymer that is soluble in water at elevated temperatures and does not reprecipitate upon cooling. The intrinsic viscosity of this polymer is reported to be 1.25 dl/g in water at 30°C. Using a relationship reported for polyacrylamide, the authors calculated a viscosity average molecular weight (Mv) of 2.4×10^5.

Titkova[*] reported that PMAAm with Mw < 6.8×10^5 is soluble in water at 25°C, and that PMAAm with 6.8×10^5 < Mw < 6.0×10^5 is soluble in water at 80-90°C, "the process being reversible," and PMAAm with Mw above 6.0×10^5 is insoluble in water at any temperature. The results of the present work agree with those of Titkova: The polymer synthesized with Mw = 2.3×10^5 fits into the second category, being soluble in water above 80°C, and the polymer with Mw = 8.1×10^5 fits into the third category delineated by Titkova, being

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insoluble in water at any temperature. However, the polymer synthesized by Matsuda and co-workers[1] with a reported molecular weight (Mv) of 2.4×10^5 would not be expected to remain dissolved in water when cooled to room temperature, based on the work of Titkova.[6]

Infrared spectra of the PMAAm homopolymer and copolymers were measured with a Perkin-Elmer 683 IR Spectrophotometer. Approximately 1 μm thick films of the resists were spin-coated onto 3-in. diameter silicon wafers, and transmission through the resist and wafer was measured using a blank wafer in the reference beam. Differential scanning calorimetry was conducted with a Perkin-Elmer DSC-4, with a scanning rate of 10°C/min. in a nitrogen atmosphere.

For lithographic evaluations, films of each resist material were spin-coated onto 3-in. diameter silicon wafers. A solution of 40% acetic acid was used for casting the PMAAm homopolymer and the 71% MAAm copolymer of MMA. The 49% MAAm copolymer was cast from glacial acetic acid, and the 21% MAAm copolymer from 2-ethoxyethanol (cellosolve). PMMA (for reference) was cast from chlorobenzene. The copolymer films were prebaked at 200°C for 15 minutes. The PMAAm homopolymer was prebaked at 180, 200, 220, and 240°C for 15 minutes for an analysis of the effect of prebake conditions on lithographic performance. Film thicknesses were measured with a Tencor alpha step.

Lithographic test patterns were produced with a Cambridge EBMF-II-150 using an accelerating voltage of 20 KV. Contrast and thinning were determined by measuring film thicknesses after developing 20 μm wide lines exposed at a series of 16 doses. Developing was conducted in stagnant solutions, terminated with an appropriate non-solvent, followed by drying with a stream of nitrogen. A 15 minute postbake at 150°C was used.

Dissolution rates were measured using a laser interferometer film thickness monitor (LIFTM)[6] for preliminary optimization of developing systems prior to lithographic evaluation. LIFTM was also used to study prebake effects on the PMAAm homopolymer by observing solubility rate ratios and swelling behavior. Dissolution induction periods were also observed with laser interferometry.

Results

The PMAAm homopolymers synthesized are not soluble in water at room temperature, and therefore films are cast from solutions in 40% acetic acid in water. In this respect, the procedures used in the present work differ from the previous work of Matsuda and co-workers,[1] who used only water for casting the PMAAm films.

Under all experimental conditions, swelling of lithographic patterns was observed for the high molecular weight (Mw ≈ 8.1×10^4) homopolymer, which limited the resolution of the lithographic patterns to about 1 μm (Figure 1). Swelling was not alleviated by altering the developer pH, or by increasing the prebake temperature. The lower molecular weight PMAAm homopolymer (Mw ≈ 2.4×10^3) also exhibits severe swelling as well as adhesion problems.
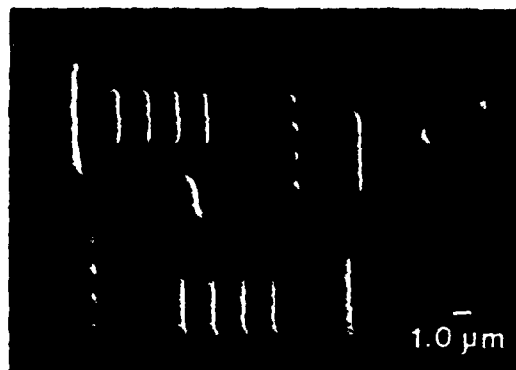

Figure 1. SEM photomicrograph of lithographic patterns written in PMAAm homopolymer. Electron dose is 20 μC/cm², and the developer is Na_2SiO_3 solution (pH = 10). The film has been prebaked at 220C for 15 minutes.
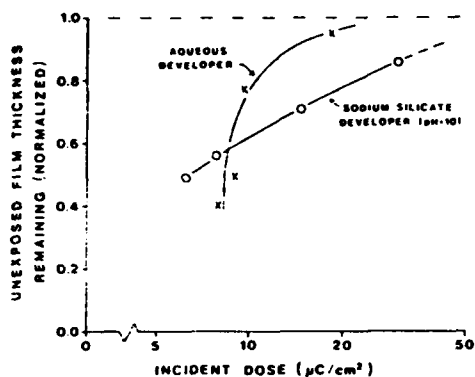
Figure 2. Thinning curves for PMAAm.
Mw = 8 x 10⁶; prebaked at 220°C. Ordinate
axis shows the thickness of the unexposed
film remaining at the time which the
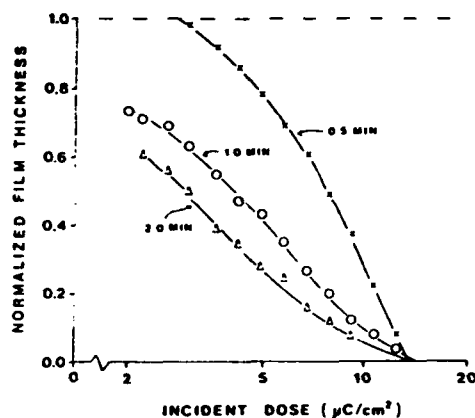dose shown on the abscissa has been
completely developed.

Figure 3. Contrast curves for PMAAm
homopolymer, 200°C prebake.

The thinning behavior of PMAAm depends greatly on the developer. Using a 220°C prebake
and developing with pure water, 10% of the unexposed background was developed in order to
completely develop a 20 μm wide line exposed at 15 μC/cm² (Figure 2). Because of the
limited solubility in water, resist exposed at doses lower than 10 μC/cm² is not developed
with pure water, regardless of developing time. Increasing developing time does not
develop lower doses, but only increases thinning of the unexposed film, presumably by
extraction of lower molecular weight fractions of the polymer. A rough calculation of
final molecular weight after exposure to 10 μC/cm² of 20 KeV electrons yields a final
molecular weight within an order of magnitude of the threshold molecular weight for
solubility in water at room temperature (Mw = 6.8 x 10⁵) reported by Titkova, et al.⁶

Dilute aqueous solutions of sodium silicate (Na₂SiO₃) are forced developers for the
PMAAm resist, and do not exhibit a solubility limit as observed with water, as even the
unexposed resist is soluble in these solutions. Thinning occurs to a much greater extent
with sodium silicate solutions than with water. Ten percent thinning occurs for
development of patterns exposed at 40 μC/cm², using a sodium silicate solution with a pH
of 10 (Figure 2). This thinning behavior in the forced developing regime does not
represent any improvement over that exhibited by PMMA.⁵

Adequate contrast is obtained when developing PMAAm with pure water. A contrast (γ) of
2.0 is measured when developing patterns exposed to a dose of 13 μC/cm² in water for 0.5
minute (Figure 3). Increasing developing time reduces the contrast and does not develop
patterns exposed at lower doses (Figure 3).

Using prebake temperatures in the range of 180 to 240°C, no significant differences in
lithographic performance are observed. Swelling occurs when using any of these prebake
temperatures, and the contrast is practically independent of prebake temperature. Prebake
temperature, over the experimental range, does not affect the solubility limit experienced
with aqueous developing, nor the degree of thinning by forced developing with sodium
silicate solutions.

Previous workers reported a sensitivity of 0.6 μC/cm² for PMAAm with an initial molecular weight (Mv) of 2.4 x 10⁶ using a 15 minute prebake at 200°C and an accelerating voltage of 20 KV.[1] Although the criterion for sensitivity determination was not published, this is clearly a greater sensitivity than that determined in the present work. What factors could be responsible for the high sensitivity previously reported for PMAAm? The chain scission efficiency (Gs), determined with gamma irradiation, has been reported to be 2.05, or about 1.5 times that of PMMA.[2] This modest increase in G-scission could not be responsible for the nearly 100-fold increase in sensitivity over that of PMMA.

It has been proposed that the sensitivity of this resist may be enhanced by an induction time during dissolution. Using a laser interferometer film thickness monitor (LIFTM)[4], induction periods are observed during dissolution of unexposed films of PMAAm and the MAAm-MMA copolymer containing 21% MAAm. The copolymer provides an excellent example of an induction period which is seen as the initial flat portion of the intensity curve that occurs before the characteristic sinusoidal output created by the interference pattern during dissolution (Figure 4). The observed induction periods are not strongly affected by exposure to vacuum or aging. In the case of the PMAAm homopolymer, the induction period is actually a period of severe swelling, occurring prior to dissolution. Since these induction periods are absent during dissolution of the exposed films, the phenomena would tend to increase the solubility rate differential between exposed and unexposed polymer, thus increasing the sensitivity. However, the magnitude of these induction periods are such that about 0.05 μm of exposed resist (20 μC/cm²) would be expected to dissolve during the induction period of the unexposed resist. The magnitude of this effect would not account for the high sensitivity reported for PMAAm resist. The concurrent swelling of the unexposed PMAAm homopolymer also limits any contribution of the induction period to enhancement of sensitivity for that system.
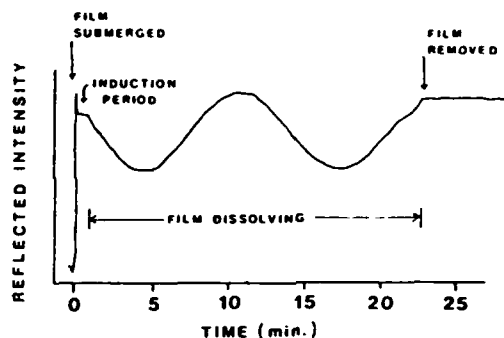

Figure 4. Dissolution induction period exhibited with poly(MMA-21% MAAm).

The most likely cause of the previously attained sensitivity reported by Matsuda and co-workers[1] is the formation of intermolecular imide crosslinks. It was reported that 62% imidization occurred after heating a 7% aqueous solution of PMAAm for 7 hours at 60°C[1] or 80°C.[3] Imide formation was also reported for prebaking on the wafer at 200-250°C.[1] Crosslinking has been shown to enhance sensitivity by increasing the initial molecular weight of resists and reducing thinning of unexposed resist.[3] Presumably because of the imide bridges, the highly selective aqueous developing systems were able to develop the relatively large exposed areas without much concurrent thinning of the unexposed resist. Under the experimental conditions of the present work, however, conversion of the amide to imide is not observed. After heating an aqueous solution of the low molecular weight PMAAm (Mw = 2.4 x 10⁵) for 7 hours at 80°C, infrared spectral analyses do not indicate the formation of imide. Also, imidization is not detected after prebaking PMAAm on silicon wafers at 180 to 240°C. Imide formation with PMAAm apparently is difficult to control. This difficulty has been encountered with other crosslinking resist systems,[8,9] and severely limits the process latitude of a resist system. Further work to reproduce and control the imidization reactions of PMAAm would be useful.

Because of the various difficulties encountered with the PMAAm homopolymer, a series of copolymers with methyl methacrylate were synthesized. Copolymers of MAAm with styrene, barium acrylate and methylmethacrylate have been studied previously, but the compositions of these were limited to copolymers containing at least 90% methacrylamide.[5] Dissolution rate measurements with the MMA-MAAm copolymers using laser interferometry indicated that the greatest solubility rate ratios were exhibited by the copolymer containing approximately 21 mol % MAAm. In contrast to the MAAm homopolymer, this copolymer did not exhibit severe swelling problems when writing 1 μm patterns. Using a 15 minute prebake at 200°C and developing with 2-ethoxyethanol, adequate contrast (γ = 2.1 at 20 μC/cm²) is observed with this copolymer (Figure 5). Unfortunately, this copolymer resist is subject to considerable thinning and in this way is only a marginal improvement over PMMA (Figure 6).

Conclusions

The sensitivity of the PMAAm homopolymer resist is about 15 μC/cm², where sensitivity is defined as the incident electron dose that can be completely developed with less than 10% thinning of unexposed resist. This sensitivity has been observed using prebake temperatures ranging from 180 to 240°C and an accelerating voltage of 20 KV. Patterns exposed at doses less than 10 μC/cm² could not be developed with water, but dilute solutions of sodium silicate developed lower doses (with extensive thinning). Swelling and adhesion problems limited the resolution of the PMAAm resist. The previously reported high sensitivity of PHAAm[1] was most likely due to the formation of imide crosslinks. Such imide formation is not observed in the present work. Imide formation is difficult to control with this resist system, which seriously limits the process latitude of the resist.
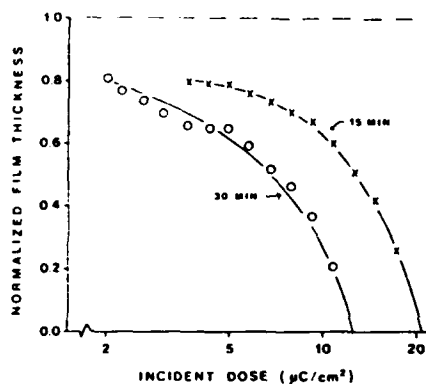

Figure 5. Contrast curve for P(MMA - 21% MAAm), prebaked 15 minutes at 200°C, and developed with 2-ethoxyethanol.
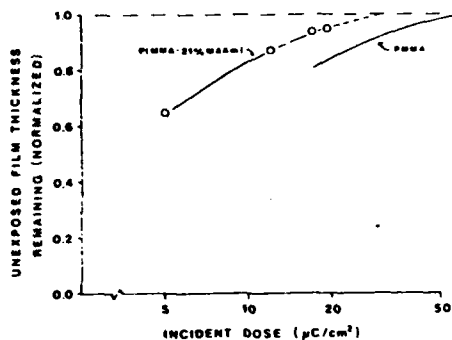
Figure 6. Thinning curve for P(MMA - 21% MAAm). The film was prebaked 15 minutes at 200°C, and developed with 2-ethoxyethanol.

Acknowledgment

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